Standard Operating Procedure

for

Routine Operation of the Climet SPECTRO 0.3 Optical Particle Counter in CRPAQS

DRAFT
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1. Scope and Applicability

This applies to the operation of the CRPAQS particle sizing system, configured to run only the Climet Instruments model SPECTRO 0.3. This system measures the ambient particle size distribution, defined as the number concentration of particles as a function of particle diameter.

2. Summary of Method

2.1. Method Parameters

The Climet SPECTRO has multiple size bins that span the measured size range and outputs the number concentration for each size bin. The parameters for the SPECTRO, as operated for CRPAQS are listed below.

Measured Parameter: Particle number concentrations units of $\#/cm^3$ per size bin.

Size Bins: 16 channels from 0.3 to 10 µm

Time Resolution: 5 min

Sample flow rates: 1 L/min

2.2. System Overview

The Climet Instruments model SPECTRO 0.l measures the large particle size range, 0.3 to 10 µm. The SPECTRO is an optical particle counters (OPC). Its sizing is based on amount of the light scattered by individual particles and is dependent on particle refractive index as well as size.

The selection of the Climet for large particle sizing was based on laboratory evaluation and comparison of several candidate instruments. Indeed, in the first evaluation none of the candidate instruments were deemed acceptable (see Appendix A in this SOP document). Two of the instruments were modified by the manufacturer based on input from Aerosol Dynamics, and retested. Of these the Climet 0.3 operated at a sample flow of 1 L/min was considered the most suitable for this study because it did not show false counts at large particle sizes, and is relatively insensitive to particle refractive index (see Appendix B in this SOP document).

The inlet and the operational principal of the instrument are described in Sections 2.3 and 2.4.

2.3. Optical Particle Counters

The OPC determines the size of a sampled particle by the quantity of light scattered by the particle and focused on to a photodetector using a system of mirrors. Since the amount of light scattered from a particle is a strong function of its size, precise and repeatable sizing of particles is possible. Particle concentrations are kept low enough within the measuring volume of the counter to insure only one particle is measured at a time. The indicated size by an OPC depends on a particle's refractive index in addition to its size. An appropriate optical calibration for use in the field is therefore required for accurate sizing of ambient particles.

2.4. Inlet

The sampling inlet system for the particle sizing instruments is designed to ensure representative sampling. The different particle size ranges measured by each instrument impose correspondingly different requirements, and these are taken into account in the design. First, it is necessary that there be no size bias in the size of particles aspirated into the sampling line over the entire size range from 0.01-10µm. Second, the transport lines to the Climet are designed to minimize losses for the specific size range covered by the instrument.

The inlet system for the CRAPQS particle sizing system is shown in Figure 1. Essential components of the inlet system are:

- PM_{10} inlet.
- Straight flow path for the Climet (large particle size range).

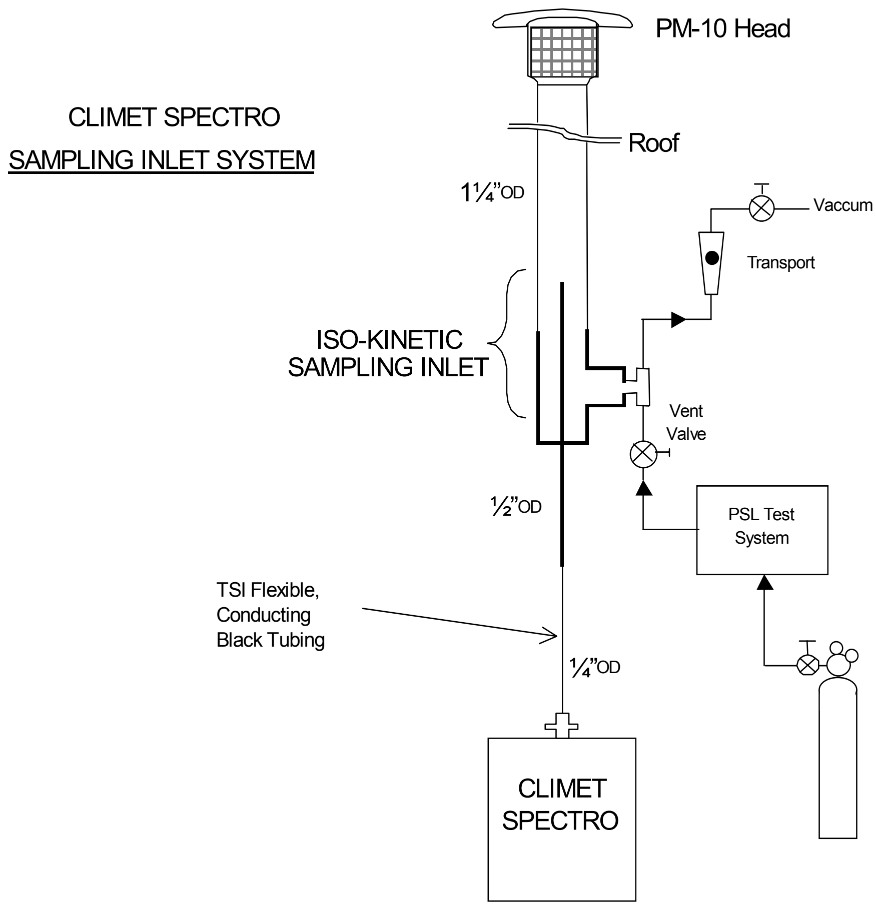

Figure 1. Sampling inlet system for particle sizing instruments.

The PM_{10} inlet was needed to provide representative sampling of coarse particles from the atmosphere, regardless of ambient wind speeds. Without this inlet, the efficiency with which particles entered the sample line would depend on wind speed, and could vary with sampling location as well as particle size. The inlet was essential for comparison of coarse particle size distributions measured on the tower sites to those measured at ground level. The sample line to the Climet was made to be straight as possible, with an isokinetic flow split, to ensure representative sampling of the particles that penetrate the PM_{10} inlet.

3. Definitions

- ADI: Aerosol Dynamics, Inc.
- CRPAQS: California Regional Particulate Air Quality Study
- OPC: Optical Particle Counter
- PM_{10} inlet: A standard precut device to select particles of less than 10-µm diameter
- PSL: Polystyrene Latex

4. Health and Safety Warnings

None.

5. Cautions

None.

6. Interferences

None.

7. Personnel Qualifications

The system requires a technically experienced operator who understands the system, its operation and calibration. This operating procedure assumes the operator can properly use flow standards and is familiar with computer operations.

8. Apparatus and Materials

Inlet consisting of:

- PM_{10} inlet
- downtube
- plumbing and electrically conducting flexible tubing
- bypass flow line with rotameter and pump

Climet SPECTRO 0.3

PSL Calibration system consisting of:

- compressed dry air source (such as cylinder of dry air)
- nebulizer setup, with metered dilution and nebulizing air flows

Supplies as listed in Table 1.

Table 1. Field Operations Supply List

1 Four latex particle sizes in 15 ml bottles from Interfacial Dynamics (prices range from $85-135 per):

Dp (µm)	Product #	Batch #	Order 1 of each size from:
			Interfacial Dynamics Corp.
0.576	1-600	684,3	17300 SW Upper Boones Ferry Road, Suite 120
0.885	1-900	10-200-60,1	Portland, OR 97224
1.418	1-1400	928,1	Ph: 800-323-4810
4.6	1-4500	736,3	Fax: 503-684-09559

2 5 dozen Devilbiss Micro-Mist Disposable Nebulizers
Product # 4650D-620 — Available from many medical supply houses
Mail order: Shield Healthcare (800-675-8840)

3 Dry compressed air cylinder and regulator
Cylinder of dry air (size 1A) for CG-590 regulator. Note -- medical air takes a different regulator, you don't want this. — Order from compressed gas distributor near site

4 Glassware: 10 ml graduated cylinder (2 ea.), 50 ml beakers (5 ea.)

5 Q-tips, Kimwipes, alcohol and distilled water bottles, cleaning detergent

9. Site and Equipment Preparation

9.1. General Setup

The site should be prepared with PM_{10} inlet and sampling system as shown in Figure 1. The Climet is situated directly underneath the downtube and installed in accordance with its manual. It requires a data line to the data acquisition computer.

10. Instrument Calibration

10.1. Overview

The Climet SPECTRO requires two types of calibration in the field: (1) flow checks and (2) particle sizing checks. The flow checks should be done routinely and are described in Section 11.

Periodic sizing calibration checks with polystyrene latex (PSL) spheres are necessary to insure instrument response does not change. These tests are only ‘spot checks’, designed to catch significant changes in sizing that would accompany such conditions as obstructed orifices, failing laser light intensities or extreme deviations from flow set points. A selection of PSL sizes have been chosen to fall predominantly in a single OPC bin for unambiguous identification of instrument response. Each PSL size is nebulized and sent to the SPECTRO (Figure 2). The peak responses are to be recorded in the particle sizing system calibration logbook.

10.2. Particle Sizing Calibration Materials

Materials for calibration are, as follows:

- Concentrated PSL stock (~8% by volume) in sizes 0.58, 0.89, 1.4 and 4.6 µm.
- Distilled water in a squirt bottle.
- 10 ml graduated cylinder.
- 50 ml beakers.
- Particle sizing system calibration (PSC) logbook.
- Nebulizers for each particle size.
- Compressed, filtered, dry air.
- Nebulization and dilution system.

Commercial sources for these items (except dry air and nebulization system) are listed in Table 1. The nebulization system is supplied by Aerosol Dynamics.

10.3. Nebulization System

A small flow nebulizer is used to generate aerosolized PSL particles. This is connected to the system as shown in Figure 2. The flow to the nebulizer is regulated by a 0-2 L/min rotameter (note the scale in units of cc/min). The nebulizer output is mixed with dry air in a mixing chamber or plenum prior to supplying the instruments. This dilution flow is regulated by a second rotameter (0-10 L/min). Excess flow is dumped through a valve into the transport flow line used with the inlet system. This vent valve must be open during PSL testing and closed during normal sampling.

10.4. PSL Calibration Procedure

10.4.1. Overview

Calibrations are done at 4 PSL particle sizes, as listed in Table 2. To calibrate the system with PSL, proceed as follows:

- Prepare the nebulization solutions.
- Take the instrument off line, with proper notation in the logbook or data system.
- Put the system into the calibration configuration shown in Figure 2.
- Run the calibration check for the particle sizes, as listed in Table 2.
- Record the results for that size.
- Run the calibration check for the next particle size.
- Return the system to operational mode.

Details for each step are given below.

10.4.2. Preparation of Solutions

Prepare the nebulization solutions in advance of calibration as follows:

- Prepare 4 nebulizers by rinsing them out with distilled water.
- Label each nebulizer with the PSL size using either a Sharpie permanent marker directly on the unit or by using suitable tape.
- Using Table 2 as a guideline, prepare each PSL solution by dispensing the given number of drops from the stock bottles directly into a graduated cylinder.
- Fill the cylinder with distilled water up to 5 or 10 ml line depending on desired concentration, and then pour the solution into a clean beaker.
- Add necessary additional distilled water to achieve the desired concentration.
- To thoroughly mix the solution, pour back and forth between the cylinder and the beaker. (Note: this formulation of PSL possesses special surface properties that avoid agglomerates without the use of sonication.)
- Pour the prepared solution into a clean, dry nebulizer chamber. Insure that the green insert is in place. Screw the nebulizer top firmly in place.
- Set the nebulizer aside and prepare the next solution. Thoroughly rinse glassware between preparations. Do not use paper towels or Kimwipes for drying as they may shed particles. Because concentrations are approximate, merely shaking out cleaned glassware is sufficient.
- If no ultrasonic cleaner is available for cleaning nebulizers between calibration checks, then always use new units each time, discarding used nebulizers after use.

10.4.3. Instrument Setup

- Record the start time for PSL calibration in the site log and the PS calibration logbook.
- Temporarily disconnect the SPECTRO from the data system by removing the serial line connector at the rear of the instrument.
- Reset the sample period to 1 minute.

10.4.4. Calibration Check Procedure

- Set compressed gas cylinder regulator to 10 PSIG.
- Open the vent valve located at the sampling inlet/transport flow split.
- Attach a nebulizer with a prepared PSL solution.
- Turn on the dilution flow in the range 4-5 L/min.
- Turn on the nebulizer supply flow in the range 1.5-2 L/min. Note: variations in nebulizer manufacturing lead to this range of operation. General rule of thumb: dilution flow is twice the nebulizer flow.
- Allow for aerosol sampling to stabilize with new PSL size, gauging from real-time indications in channel response.
- Record a minimum of ~5,000 counts total that fall in the peak channel (over multiple sampling periods, if necessary). Increase nebulizer flow by 0.2 L/min increments until concentrations fall within expected ranges given in Table 2.

- Print a representative sample with the instrument's built-in line printer. Accumulate printouts for all tests prior to removing from printer, then paste into calibration logbook.
- Record in log status sheet whether or not peak response falls in correct channel.
- Turn off nebulizer flow and repeat for all sizes.

10.4.5. Calibration Shutdown Procedure

- Turn off the nebulizer and dilution flows.
- Close the vent valve.
- Reconfigure the normal sampling lines with the original sampling tubing.
- Reconnect the SPECTRO to the data system.
- Record the time back online in the site logbook.
- Check to verify that the SPECTRO is sending data to the CRPAQS Data Acquisition System normally. If not, cycle the power.
- Perform the daily checks (Section 11.1) to verify that all parameters are within acceptable limits.

Table 2. PSL test parameters

PSL size D_p (µm)	# Drops PSL stock	Volume Distilled Water (ml)	Approximate[a] Conc. (#/cm^3)	Peak Bin SPECTRO
4.6	5	5	2	12
1.4	2	5	4	7
0.89	2	5	60	6
0.58	1	10	200	4[b]

Table notes:

[a] Concentrations are only approximate owing to nebulizer and flow rate variability.
[b] Instrument response should be within 1 channel of these values.

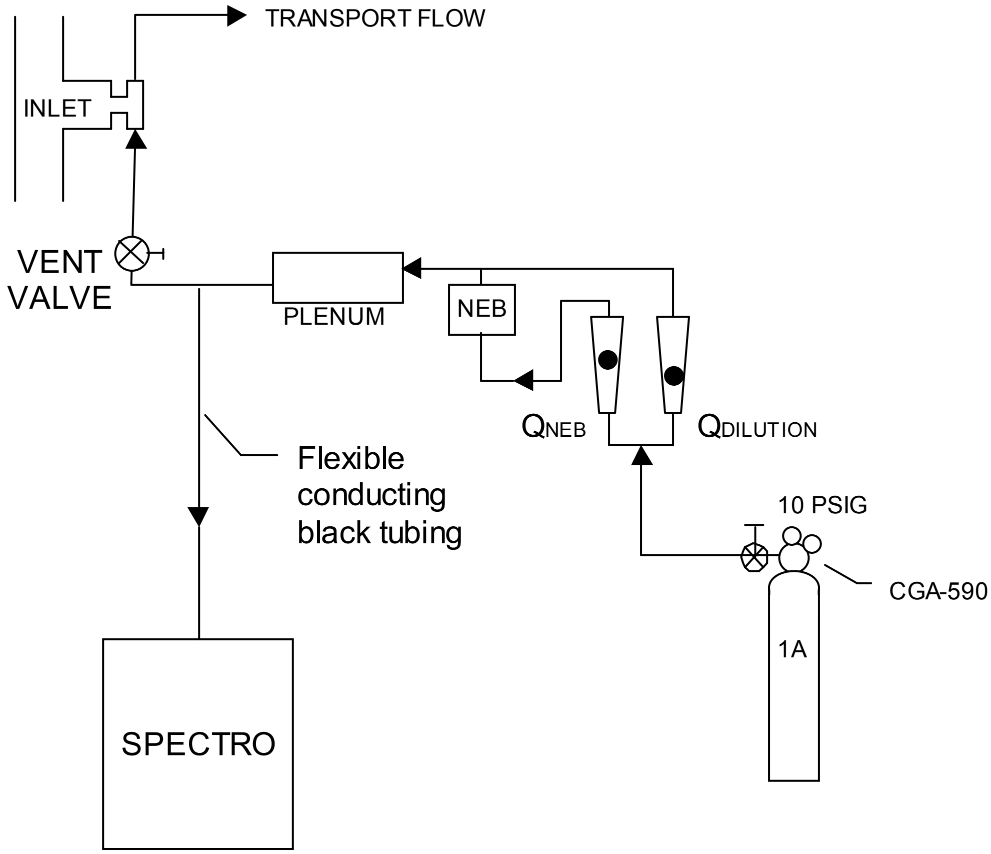

Figure 2. PSL test configuration.

11. Instrument Operation

11.1. Daily Checks

Operating conditions and OPC status should be checked daily. All Climet SPECTRO parameters can be viewed from the main display when the '**SMALL**' display setting is in effect (keypad sequence: **DSPL|SIZE,HIST**, then use up/down arrows to select '**SMALL**'). See page 3-5 of Climet manual for an example display screen.

- Check to verify that the instrument's front panel display corresponds to the allowable parameter values given in Table 3.
- Record instrument's flow rate and any out of range parameters on site log.
- Verify that total counts in the lowest bin (0.3-0.4 µm) are on the order of $1x10^6$ for a 285-second sample interval. The operator should note typical count values during normal operation for future reference.
- Examine the CRPAQS Data Acquisition System screen to verify that the instrument's data is being properly stored. Logged values include flow rate in L/min and 16 channels of counts.

Table 3. Acceptable Climet SPECTRO 0.3 Parameters

PARAMETER	DISPLAY	ALLOWABLE VALUES
Mode	DIFFCOUNT	DIFFCOUNT
Flow rate	FLOW= 1.0 L/min	0.95-1.05
Battery charge	CHARGE=>3.0 HR	>1
Sample program	PROGRAM=CRPAQS	CRPAQS
Sample Period	SAMP TIME=285	285
Sample start delay	DELAY=00:00:00	00:00:00
Sampling mode	#SAMPLES=1	1
Multiple configurations	CLASS= OFF	OFF

11.2. Monthly Checks

11.2.1. Flow check

The flow rate calibration should be checked at least once per month. Necessary equipment includes a primary flow standard such as a BIOS flow meter and assorted plumbing.

- Using a short segment of flexible tubing, connect the SPECTRO inlet to the BIOS outlet.
- Take an average of 10 readings with the BIOS for each instrument. Note: BIOS readings perturb the mass flow readings on the SPECTRO's front panel but do not significantly impact the accuracy of the flow measurements. Therefore, ignore the fluctuating panel readings while the BIOS is in use.
- Record the average flow rates in the site log.
- If the instrument fails to give an average volumetric flow rate within the limits given in Table 3, recheck flow to verify problem is persistent before proceeding to Section 15, Troubleshooting.

11.2.2. Leak check

The plumbing integrity should be checked at least once per month. Necessary equipment includes a HEPA capsule filter and assorted plumbing.

- Attach a HEPA filter capsule to the 1/4" OD inlet of the SPECTRO.
- After sampling for approximately 5 minutes, manually begin a 1-minute sample and verify that no more than 1-2 counts appear (always in the lowest channels) during this period.
- Record zero count in the site log.
- If more than 1-2 counts/min are measured, then repeat several times to verify problem is persistent before proceeding to Section 15, Troubleshooting.

11.3. Restart Procedure

On power interruptions the instrument will retain all necessary sample parameters and resume sampling at the next 5-minute mark of the CRPAQS Data Acquisition System. Steps outlined under Section 11.1, Daily Checks, should be executed after each power failure to insure that the instrument resumes normal operations. Note that a power loss less than the remaining battery charge of the SPECTRO will result in uninterrupted operation of this instrument.

12. Handling and Preservation of Samples

Not Applicable.

13. Sample Preparation

Not Applicable.

14. Preventative Maintenance and Repairs

Maintenance issues for the duration of CRPAQS are addressed through the operational checks described in Section 11. For longer term operation one should clean the PM_{10} inlet. The SPECTRO may need cleaning of its optics and recalibration by the factory if their performance is not within operating specifications.

15. Troubleshooting

15.1. Flow Rate Errors

If the SPECTROS's flow rate falls outside the allowable range, then the following steps should be taken:

- Verify that tubing connections between the BIOS and instrument are snug.
- Perform a second flow check. If available, use a different flow meter. If readings are low, conduct an internal leak check on BIOS.
- Determine if the SPECTRO reports actual flow rate or internal readings are in error.
- If actual flow rate is in error for the SPECTRO, consult manufacturer Climet Instruments for possible return to factory for service.
- If internal flow rate readings are in error but the actual flow rate drawn by the instrument is within the acceptable limits, then merely note this discrepancy in the site log and return instrument to normal operation.

15.2. Leak Check Errors

If unacceptably high levels of counts are recorded during leak checks, then the source of leakage should be determined before consulting with the Climet Instruments.

- Verify that high zero count is not a result of a contaminated filter capsule (e.g., one that was connected in reverse flow order) by use of a second filter.
- While performing a zero count check pinch off the sheath flow tubing (connects the external pump located on the back of the unit to the inlet block) to determine if the external pump is the source of particles. If no reduction in zero counts results, then the leak is most likely internal to the instrument.

- If all zero counts fall within the lowest channel, then electrical noise may be the source. Disconnect the RS-232 lines and relocate instrument away from sources of electrical interference and re-perform the leak test. If zero counts are reduced to acceptable levels, then electrical noise is the source.
- If unacceptable zero count readings persist, consult the manufacturer with results of the above tests.

15.3. Instrument Manuals

Besides the steps outlined in Sections 15.1 and 15.2, operators should consult the manufacturer's instrument manual for troubleshooting guidelines.

16. Data Acquisition, Calculations, and Data Reduction

16.1. Raw Data

Data from the SPECTRO is stored in the form of differential particle counts per sample period (originally set to 4:45). Specifically, the SPECTRO counts the number of incident particles that fall within a fixed size range, a 'bin' or channel, incrementing each count as they arrive. All particles exceeding the lower boundary of the top channel are recorded in that 'oversize' bin, therefore size distributions are only obtainable up to this bounding size (10 µm for the SPECTRO as PSL equivalent sizes).

16.2. Reduced Data

To report the data in a form that can be readily compared to other instruments requires converting the raw counts to particle number concentration or particle volume concentration as described in this guide. To calculate a concentration, the particle counts must be divided by the sample volume for each data record:

$$n_i = \frac{N_i}{V_s}, \quad (1)$$

where N_i is the number of particle counts in the i^{th} bin, n_i is the concentration in the i^{th} bin in units of cm^{-3}, V_s is the sample volume $= Q \times \Delta t$, Q is the flow rate in cm^3/min and Δt is the sample period in minutes. Conversion of flow rate to volume is necessary for the SPECTRO since this instrument only records average flow rate during each sample period.

Comparisons to be made with other instruments usually require reducing the size distributed OPC counts into a total particle concentration over some size range comparable to a size range of another instrument. Total or subinterval concentrations are obtained by simple summation of n_i:

$$n = \sum_{i\min}^{i\max} n_i, \quad (2)$$

where $i_{\min}$ and $i_{\max}$ are the appropriate channel numbers corresponding to the desired particle diameter limits (see Section 16.3, Optical Calibrations).

For instance, to compare total concentrations between the SPECTRO, n_{SPEC}, and the LASAIR, n_{LASR}, the following summations should be used:

$$n_{\mathrm{SPEC}} = \sum_{1}^{7} n_{i,\mathrm{SPEC}}\ , \quad n_{\mathrm{LASR}} = \sum_{3}^{7} n_{i,\mathrm{LASR}} \tag{3}$$

This comparison assumes that the original manufacturer's calibration was in use for the Climet SPECTRO. The PMS LASAIR channel boundaries are non-adjustable.

For comparisons to non-particle counting instruments, e.g., nephelometers or mass measurement instruments, conversion of OPC number concentrations to volume concentrations is desirable. Once an appropriate particle diameter range has been selected based on an appropriate optical calibration (see next section) the following relations may be used to compute aerosol volumes:

$$\mathrm{V_i} = \frac{\pi}{6}(\overline{\mathrm{D_p}})^3,\ \overline{\mathrm{D_p}} = \sqrt{\mathrm{D_{p,j+1}} \times \mathrm{D_{p,j}}}\ , \tag{4}$$

where V_{i} is the mean particle volume in μm^3 corresponding to the geometric mean particle diameter, $\overline{D_{\mathrm{P}}}$, of the i[th] bin with bounding particle sizes of $D_{\mathrm{P},i}$ and $D_{\mathrm{P},i+1}$. The particle volume concentration, V, in units of $\mu m^3/cm^3$ is obtained by multiplying V_i by the particle number concentration, n_i, and summing over the desired size range:

$$V = \sum_{i\min}^{i\max} n_i \times V_i \tag{5}$$

16.3. Optical Calibrations

Use of an appropriate optical counter calibration is necessary for accurate size distribution measurements. Ideally, the response of an optical counter to varying changes in ambient particle conditions would be measured routinely (Stolzenburg and Hering, 1998). Alternatively, an 'average' optical state for the ambient aerosol under consideration can be used in the form of suitable laboratory calibrations using aerosols with appropriate refractive indices, e.g., oleic acid or dioctyl sebacate (McMurry and Hering, 1989; Stolzenburg and Hering, 1998).

Table 8 is Climet Instruments factory calibration of the SPECTRO 0.3 optical counter using multiple sizes of monodispersed polystyrene latex particles. During in-house experiments at Aerosol Dynamics, this PSL calibration was found to produce close agreement of the SPECTRO and an Aerodynamic Particle Sizer (TSI model 3320) when subjected to oleic acid or Berkeley ambient aerosol (see Appendix B in this SOP document).

Table 8. SPECTRO 0.3 PSL Calibration

Bin	Dp,lo	Dp,hi	dlnDp	GM(Dp)	Volume
1	0.30	0.40	0.2877	0.346	2.18E-02
2	0.40	0.50	0.2231	0.447	4.68E-02
3	0.50	0.63	0.2311	0.561	7.55E-02
4	0.63	0.80	0.2389	0.710	1.25E-01
5	0.80	1.00	0.2231	0.894	3.07E-01
6	1.00	1.30	0.2624	1.140	7.76E-01
7	1.30	1.60	0.2076	1.442	1.57E+00
8	1.60	2.00	0.2231	1.789	3.00E+00
9	2.00	2.50	0.2231	2.236	4.83E+00
10	2.50	3.20	0.2469	2.828	8.88E+00
11	3.20	4.00	0.2231	3.578	2.18E+01
12	4.00	5.00	0.2231	4.472	4.68E+01
13	5.00	6.30	0.2311	5.612	7.55E+01
14	6.30	8.00	0.2389	7.099	1.25E+02
15	8.00	10.00	0.2231	8.944	3.07E+02
16	10.0	–	–	–	–

Legend for Table 8

Dp,lo = lower bin boundary in µm
Dp,hi = upper bin boundary in µm
dlnDp = $\log_e(D_{p,hi}) - \log_e(D_{p,lo})$
$GM(D_p)$ = geometric mean diameter = $SQRT(D_{p,hi} \times D_{p,lo})$ in µm
Volume = $(\pi/6)(GM(D_p))^3$ in μm^3

17. Computer Hardware and Software

The SPECTRO is connected to the site Data Acquisition System with a serial line. It does not require any other computer hardware or software.

18. Data Management and Records Management

As part of daily maintenance, data records should be examined to ensure that there are no significant deviations in any parameters or values. Data files collected by the Data Acquisition Systems will be stored electronically by Sonoma Technology, Inc. Log books and log sheets will be stored and reviewed during data analysis.

19. References

Hering, S.V. and McMurry, P.H., Response of a PMS LAS-X laser optical counter to monodisperse atmospheric aerosols, *Atmos. Environ.* 25A:463-488 (1991).

Stolzenburg, M. R. and Hering, S. V., A new method for the automated high-time resolution measurement of $PM_{2.5}$ nitrate, in *$PM_{2.5}$: a Fine Particle Standard*, J. Chow and P. Koutrakis editors, *J. Air Waste Management Assoc*, pp 312-317 (1998).